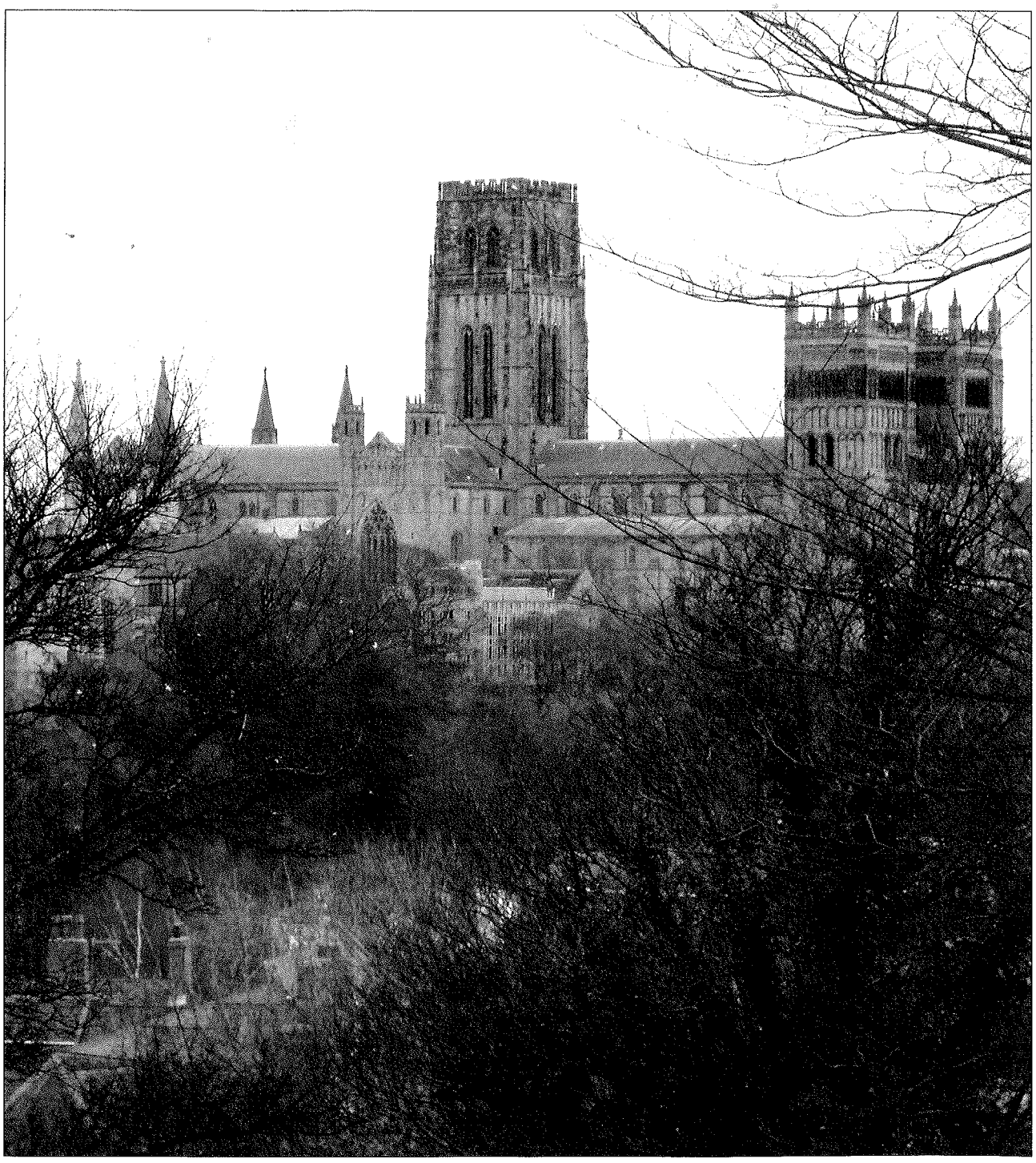
Volume 19 2012

The Journal of the Building Limes Forum

To encourage expertise and understanding in the use of building limes



Characterization of binders in the historic lime mortars and plasters from 1A Royal Crescent, Bath

Steffan Raw-Rees, Giovanni L. A. Pesce, Richard J. Ball, Enrico Fodde

Staffan Raw-Rees (MEng) graduated in Civil and Architectural Engineering from the Department of Architecture and Civil Engineering, University of Bath, in 2012. He currently works for Mace Group as a trainee graduate engineer.

Giovanni L. Pesce (MA, PhD) is a researcher in the BRE Centre for Innovative Construction Materials, Department of Architecture and Civil Engineering, University of Bath. He frequently acts as a consultant specialist for private and public research groups, institutions, and private companies in the field of built heritage. In 2010 he became a member of the editorial board of the *Open Journal of Archaeometry*, and acts as a specialist for material sciences and architecture.

Richard J. Ball (BEng, PhD, CSci, CEng, MIMMM) is a lecturer in the BRE Centre for Innovative Construction Materials, Department of Architecture and Civil Engineering, University of Bath. His research interests have focused on microstructure/macrostructure property relationships, setting reactions, degradation processes, and water transport mechanisms in lime-based binders.

Enrico Fodde (MA, PhD) is a lecturer in the BRE Centre for Innovative Construction Materials, Department of Architecture and Civil Engineering, University of Bath. He was formerly international project director of Moenjodaro (World Heritage Site, Pakistan), consultant to the Abu Dhabi Authority for Culture and Heritage (UAE), and field director for a number of UNESCO projects.

There is a growing interest in the UK building industry in gaining further knowledge of historic mortars. In 2011, permission was given by the Bath Preservation Trust to collect samples of historic lime mortars and plasters from 1A Royal Crescent, Bath. A total of nineteen were collected for characterization from various locations inside the building and grouped into several mix types following preliminary analysis. A representative sample from each group was then chosen for further investigation with optical microscopy at low and high magnification, scanning electron microscopy (SEM), energy dispersive analysis of X-rays (EDX), X-ray diffraction (XRD), and differential thermal analysis / thermal gravimetric analysis (DTA/TGA). After a visual inspection of the established groups, a detailed analysis of the pure binder of the mixes was carried out using pure lime lumps extracted from the specimens. Analysis of the binder was well defined and all the analyses carried out were in agreement. The lime binder contained 64% of calcium carbonate together with some hydraulic compounds and quartz due to the aggregate. Results established that the binder was a natural feebly hydraulic lime and provided useful information as required to specify a compatible repair mortar.

Historical overview

The city of Bath is located in south-west England. It was inscribed in the UNESCO World Heritage List in 1987, with the justification of being 'of international importance for its contribution to the art of urban design, for its architectural quality, its Roman remains, its Georgian town centre and its historic associations'. A major contribution to the development of Bath was made in the 18th century by John Wood the elder (1704–1754), who built Queen's Square (1728–39) and the Circus (1754–60). His son, John Wood (1728–1782), followed by building the Royal Crescent (1767–74).

Number 1A Royal Crescent (Figure 1a) was built in 1765 and is listed Grade II (nationally important and of special interest), whilst the adjoining Number 1 is listed Grade I (of exceptional interest, considered to be internationally important). In 1970 Number 1 was opened as an eighteenth-century domestic museum, and it is now the seat of the Bath Preservation Trust. Despite the lower grading given to Number 1A, its importance relates to its proximity to the Royal Crescent and to the fact that it was used as a service wing for Number 1. Writer and critic George Saintsbury was the most famous resident of 1A Royal Crescent and he lived there until 1933, whilst the last private inhabitant lived there until 1967.[1] In 2006 the house was purchased by the Bath Preservation Trust, which has recently been awarded £1.4 million by the Heritage Lottery Fund to restore the building. The aim of such work is to return the building to its earlier state, the servants' quarter to Number 1, and open it together with Number 1 as a museum.[2]

Fig. 1a Number 1A Royal Crescent.

The building is constructed of Bath stone, which is a soft and porous oolitic limestone made of granular fragments of calcium carbonate.[3,4] This stone has been quarried locally since Roman times, and it was used widely for the construction of Bath.[5] The building has two main facades: the southern shows little alteration, whereas the eastern (Upper Church Street) reveals a Venetian window infilled with stonework.

Assessing historic lime mortars: the philosophical background

Current research interests have centred on the development of methods for comparing historic and repair material, particularly in the field of earthen building materials and lime mortar conservation.[6-10] Many of these studies agree that the ICOMOS philosophical principles of reversibility, compatibility, expandability, and 'like-with-like repair' are fundamental requirements when designing repair mortars. However, at present there is no standard characterization protocol for achieving such principles. Laboratory work covers a range of analytical techniques for characterizing mortar constituents and for describing mortar properties in an effective way,[11] but a methodology for studying the ICOMOS principles still needs to be developed.

Yates and Ferguson state that compatibility can be defined as the matching of repair mortar to those already in the historic building.[12] It is often recommended that the experimental analysis chosen to characterize historic mortars should provide valuable information such as

the type of aggregate and binder used, whether or not pozzolans are used, and also the hydraulicity of the lime.[13] Therefore, repair mortar must be based on the original mortar characterization. As a result it will behave similarly and will not have any negative consequences on the authentic material, such as initiating new types of damage and affecting the integrity of the structure.[14] According to Carrington and Swallow,[15] lime mortar needs to be softer and more porous than masonry. It should be added here that lime mortar used in repair needs to be softer and more porous than the historic mortar too. In so being, it behaves as a sacrificial substrate that allows water evaporation[16–20] and soluble salt weathering to take place. Furthermore it should be noted that real world conservation practice has different and more practical requirements such as aesthetic features, workability, accelerating hardening, and use of volumetric rather than mass parts.[21] The choice of analytical techniques used depends on the question that has to be answered and also on the amount of material available.[22] In any case, all information gained must be supported by combined methods.

The aim of this paper is to propose a methodology for studying binders in historic mortars which will provide information useful in defining the sacrificiality of repair binders. It should be noted that in this paper the term 'sacrificiality' does not apply to the relationship between the repair binder and the limestone with which 1A Royal Crescent is built, but rather to the repair binder in its relationship with the historic binder.

Binder characterization

Use of lime lumps in the study of historic binders

In this study, the binder characteristics were determined through investigation of the pure lime lumps. Data from these is not influenced by the additives and aggregate which could have been added to the mixes and that make interpretation of results more difficult. Lime lumps can provide invaluable information about the historic technologies used to produce a lime binder. These include initial composition of the lime, its hydraulicity, and the hardening process,[23] in addition to the age of the mix.[24–26] But in order to obtain this information it is important to know that there can be, at least, three types of lumps inside the old lime-based mixtures.

In old lime mixtures it is quite easy to see small or large rounded lumps, often characterized by shrinkage cracks.[27] Some of the lumps are considered to be partially sintered/fused particles formed in traditional kilns but other types can be derived from the carbonation of 'lumps' of lime not mixed with aggregate and additives.[28,29] Callebaut and Van Balen[30] report that the presence of large, unmixed, well-rounded porous lime lumps greater than 3 mm in size within a mortar suggests that the lime used was dry-slaked, or slaked with a minimum amount of water.[31–32] Lime lumps can also be made of under-burnt, partially burnt, or over-burnt pieces of stone,[33] and for this reason identification of the different minerals in lime can be used to estimate the burning temperature of the limestone used to produce the lime. Furthermore, under-burnt or partially burnt lime lumps often exhibit the original limestone texture, which enables the identification of the geological source of the limestone burnt in the kiln.[34,35]

Additives

Together with lime, the binding phase of old lime-based mixtures included other constituents such as hydraulic additives and animal fibres. Animal hair and straw are often found in lime-based mortars, especially in plasters and renders. These fibres were traditionally used as reinforcement to improve the tensile strength of the material, but also the mortar cohesiveness.[36,37] The best varieties of hair fibre are said to come from goat (usually white colour) and cattle (usually dark brown/reddish colour). Such fibres are covered with tiny barbs, which hold the hairs in place within the mortar.[38] Horse hair, which can come in a variety of colours, is thought to be less suitable because of its smooth surface, as a result of which it can be pulled away from the hardened mortar by hand.[39] Charcoal is often present as small inclusions in historic lime due to contamination from the fuel used in the kiln.[40,41] The presence of small, random quantities of charcoal in mortars is a legacy of the burning process. Where large quantities are evenly distributed it will have been added as a pigment to darken the mortar,[42] or to act as a pozzolan.[43,44]

Materials and methods

Sample collection

In April 2011, before conservation work began, permission was given by the Bath Preservation Trust to collect mortar and plaster samples. Nineteen specimens were removed using a hammer and chisel. Sampling was carried out in order to represent different construction phases and material was exposed to a range of different environments such as temperature and relative humidity.[45] A description of each sample is given in Table 1 and the corresponding location in Figure 1b.

Visual analysis

Visual analysis using a Leica Wild M3 stereo optical microscope with fibre optic lighting and magnification up to x 50 was carried out to identify compositional and physical characteristics of the lime samples.[46, 47] Low power microscopy provided important information

Sample number	Location	Description
S1	Room FF8	Skim coat as applied on S2, blocked window
S2	Room FF8	Scratch coat on lath, blocked window (centre right)
S3	Room FF8	Scratch coat on lath,, blocked window (top left)
S4	Room FF8	Skim coat as applied on S3, blocked window
S5	Room FF7	Scratch coat on lath, blocked window
S6	Room GF7A	Plaster under staircase of Gentlemen's Retreat
S7	Room FF8	Scratch coat, wall under the floorboards of the north wall
S8	Room above entrance hall	Coat with limewash layers
S9	Room GF6	Plaster coat of concealed wall (original wall) under the floorboards
S10	Room GF6	Plaster coat of sleeper wall under the floorboards
S11	Room GF6	Concealed wall mortar under the floorboards
S12	Room GF11	Mortar under floorboards
S13	Room GF11	Combined samples of scratch coat, skim coat, and paint layers as found under the floorboards
S14	Room B8	Mortar as found under the floorboards (Housekeeper's Room)
S15	Room B9	Plaster – west end
S16	Room B9	Skim coat – east end
S17	Room B9	Scratch coat – east end
S18	Room B10	Coat over iron element – south wall
S19	Room B11	Skim coat – north wall

Table 1 Room numbering and description of the nineteen samples collected from 1A Royal Crescent.

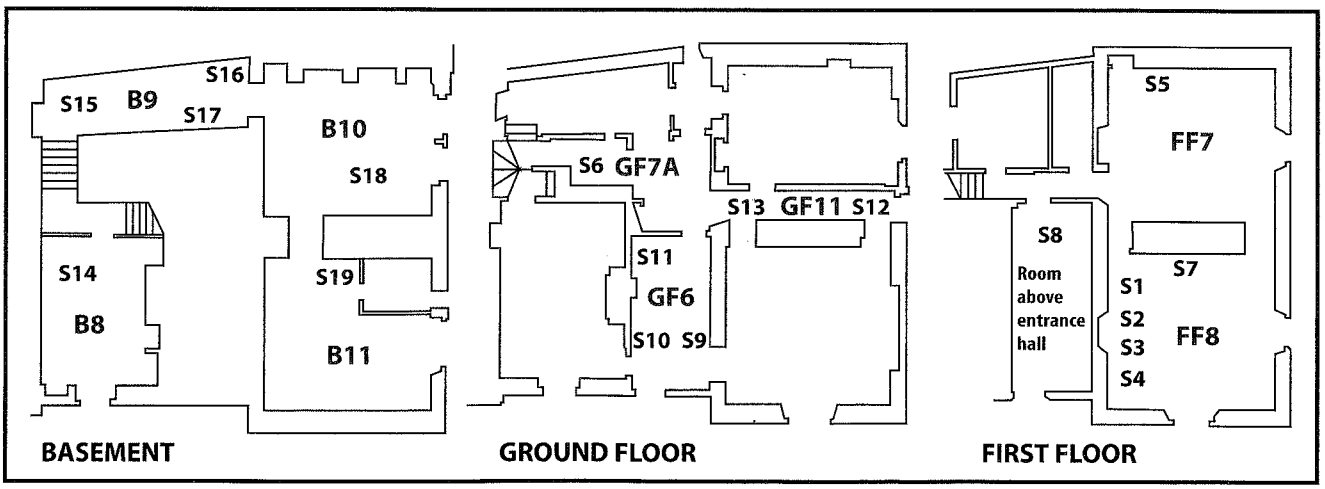


Fig. 1b Plan of 1A Royal Crescent, showing location of mortar samples.

Samples	Red brick	Carbon	Lime lump	Fibre (hair/wood)	Plaster
4, 13, 18	X	X	X	X	X
14	X	X	X	X	
17	X	X	X		
6, 19		X	X	X	X
2, 3, 5, 7, 8		X	X	X	
15		X	X		X
9, 11, 16		X	X		
1		X		X	X
12		X		X	

Table 2 Range of different mortar compositions within 1A Royal Crescent, established from the low magnification optical microscopy examination.

Sample number	Wood fibre	Hair fibre	Carbon	Lime lump	Yellow lump	Gravel	Red brick	Plaster
14	X		X	X	X	X	X	
18		X	X		X		X	X

Table 3 Compositions of Samples 14 and 18, identified following detailed examination.

such as the presence of additives in the mortar, the colour and number of plaster coatings, the grain size and shape of aggregate, and the presence of inclusions and damage.[48] Therefore similarities between samples could be found, as summarized in Table 2. A full list of phases identified in Samples 14 and 18 following a more detailed examination and analysis is given in Table 3.

Optical microscopy of polished sections

Polished cross sections were made from representative mortar fragments no more than 25 mm by 15 mm in size. Samples were embedded in low-viscosity resin by vacuum impregnation. Once set, the surface of each sample was ground and polished using progressively finer silicon carbide, diamond, and silica polishing media. Images of the polished sections were obtained using a Zeiss ICM 405 metallurgical microscope.

Micro morphological analysis with scanning electron microscope

A JEOL JSM-6480LV scanning electron microscope was used to determine the structural morphology of the samples. Polished sections of mortar fragments were prepared as described by El-Turki et al.[49] and analysed. To prevent charging all samples were stored in a vacuum desiccator for 24 hours before sputter coating with a thin layer of gold using an Edwards sputter coater S150B. Energy dispersive X-ray analysis (EDX) was used to determine the elemental composition where necessary, which proved complementary information to support the other techniques employed.[50] EDX analysis was performed using an Oxford Instruments INCA X-act detector. The analysis was performed on uncoated samples to avoid overlap of gold peaks with peaks of interest, using a higher accelerating voltage of 20 kV and a larger spot size than for imaging. EDX spectra were obtained between 0 to 10 keV; however, no peaks were identified above 5 keV and results are presented in the range 0 to 5 keV.

Thermogravimetric analysis (TGA) and differential thermal analysis (DTA)

Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) were carried out to identify phases within the mortar samples through their endothermic and exothermic reactions during heating. TGA/DTA allows accurate quantification of the presence of portlandite and calcium carbonate.[51] The technique can also identify whether the mortar is hydraulic or non-hydraulic. Furthermore, amorphous compounds and compounds with low crystallinity that cannot be determined using XRD can be characterized by using TGA.[52] Specimens chosen for the thermal analysis were carefully removed from each sample using a scalpel, and crushed into a powder using an agate pestle and mortar. Around 15 mg of the powder was placed into separate 100 µl alumina crucibles. Argon and nitrogen were passed into the equipment, which was heated at a rate of 20°C per minute up to 900°C. A white lump from Sample 14 and white plaster from Sample 18 were analysed, and the composition was then calculated as described by Ball et al.[53]

XRD analysis

X-ray diffraction (XRD) was performed on powdered samples ground in an agate mortar and held in a 700-micron-diameter glass capillary tube using a Bruker D8 Advance powder diffractometer. Analysis was performed using Cu Kα X-rays of wavelength 1.5418Å over a 2-theta range of 15 to 60° at 20°C.

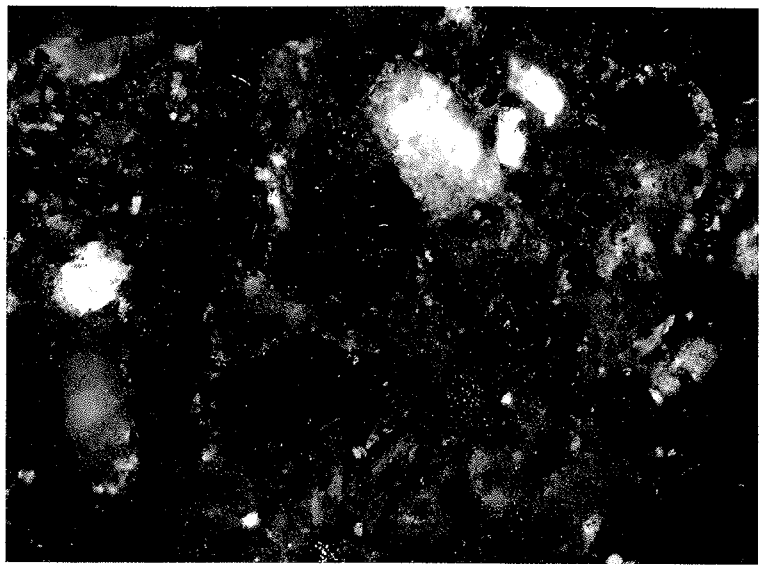
Results and discussion

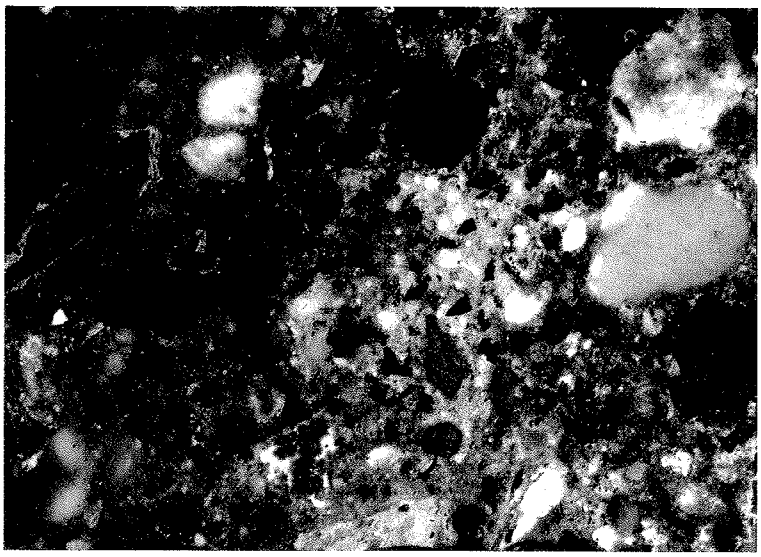
Of the nineteen specimens collected from within the building, visual analysis identified a range of different additives including red brick, carbon, white lumps, fibres, and a finishing layer. A summary of these is shown in Table 2. This paper presents results from an examination of the binder from a typical mortar (Sample 14) and plaster (Sample 18).

Examination of lime mortar (Sample 14)

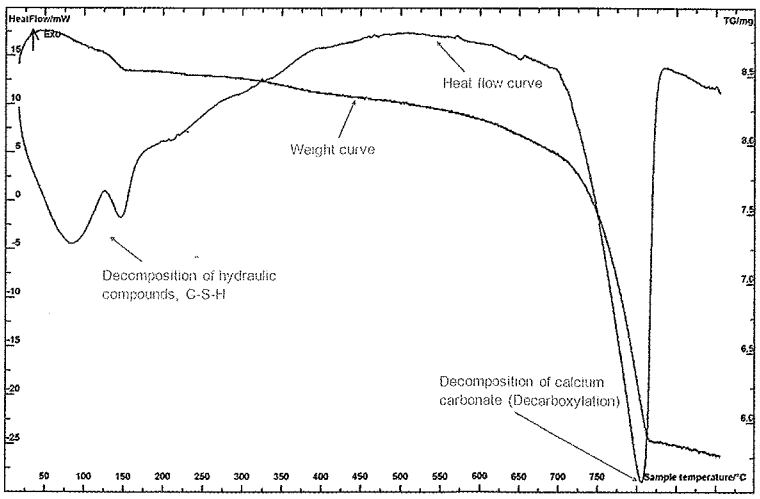
Even though no strength test was undertaken for Sample 14, the mortar was very hard and not friable during handling. This sample, moreover, together with many of the other specimens, contained a high proportion of carbon inclusions. The conjecture is that crushed carbon might have been added to the mix in order to darken the colour of the mortar, or this large amount of charcoal may be due to the use of traditional lime production techniques. In fact, it is unlikely that, centuries ago, it would have been possible to completely separate the lime from the fuel. The polished sections shown in Figures 2 and 3 show carbon (black) and lime lump particles (white), with granulometry towards the fine end of the index. This could indicate hard burning (extended time and temperature during firing). This is consistent with traditional firing techniques, in which difficulties in precisely controlling temperatures throughout the kiln are well known. These higher temperatures resulted in the formation of different hydraulic components and compositional variations.

To investigate the binder used in this mix, a lump of pure lime was extracted from the sample. The TGA analysis of the lump is shown in Figure 4. The initial increase in weight is attributed to a buoyancy effect within the furnace.[54] In the range 50 to 125°C an endothermic peak and a weight loss is observed due to absorbed water. An endothermic peak in the temperature range 650 to 850°C is characteristic of calcium carbonate. The corresponding weight loss was used to calculate the calcium carbonate content using the method described in Ball et al.,[55] which was 64%. The absence of an endothermic peak at about 500°C indicated that $Ca(OH)_2$ was not present, which is also verified by the XRD data shown in Figure 5, and indicates the material was completely carbonated. A subtle reaction can be seen within the range 125 to 150°C,





Figs 2 and 3 Polished sections showing carbon (black) and lime lump particles (white).



Figs 4 TGA analysis of the lump.

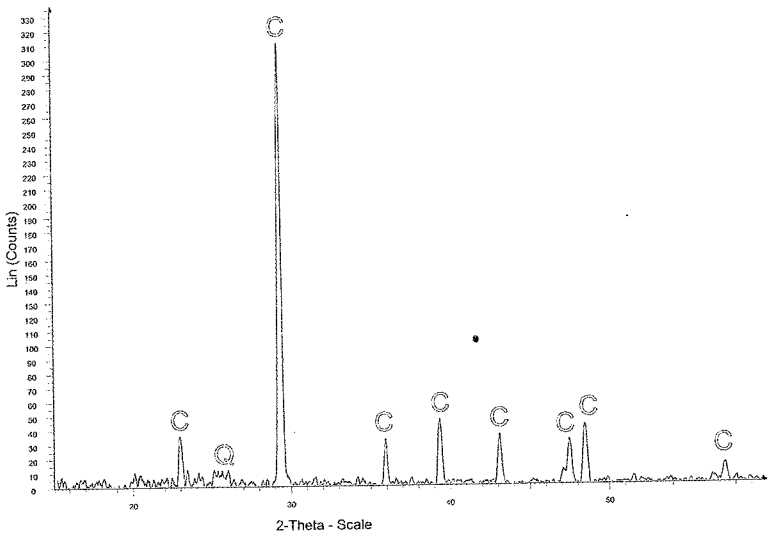


Fig. 5 EDX analysis.

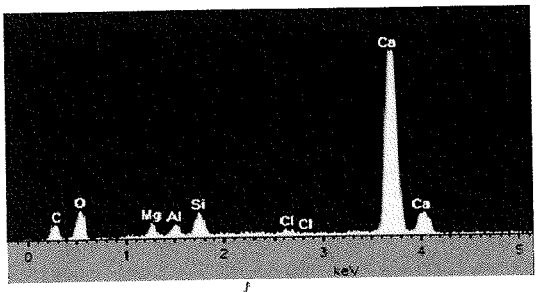


Fig. 6 EDX spectrum.

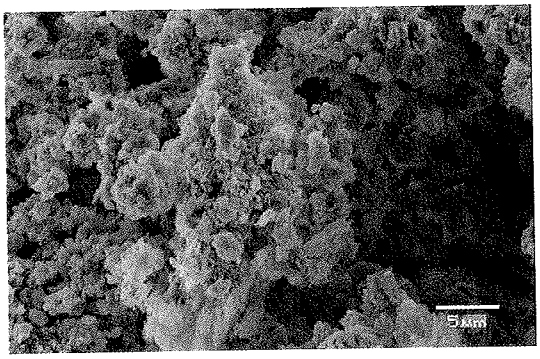
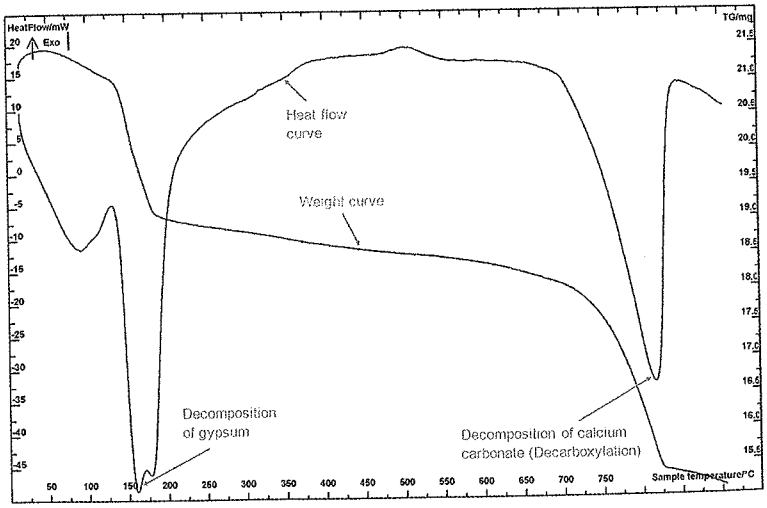


Fig. 7 SEM image suggesting carbonated microstructure.

which defines the decomposition temperature of both gypsum and hydraulic compounds such as the CSH. However, the negligible amount of sulphur detected by the EDX analysis, Figure 6, does not support the presence of gypsum and suggests the presence of hydraulic compounds. Furthermore, according to Moropoulou and other researchers,[56,57] the weight loss between 200 and 600°C appears to confirm the presence of hydraulic compounds. Moreover, the high percentage of $CaCO^3$ and the shallow gradient seen from the TGA analysis indicate that the sample was feebly hydraulic.

The XRD pattern shown in Figure 5 indicates the crystalline phases in the sample are predominantly calcite by peaks located at 2-theta values of 22.9, 29.1, 36.1, 39.3, 43.2, 47.3, 48.5, and 57.5°. The SEM image of the same sample shown in Figure 7 suggests a carbonated microstructure is present due to the angular calcite crystals observed. The peak at 2-theta 26 is attributed to the high intensity (001) reflection of silicon dioxide, quartz. This is thought to originate from the aggregate, which was also verified through observing moderate intensity peaks of silicon in the EDX spectrum, Figure 6. Randomly distributed quartz particles with angular grains are visible in the optical microscopy images shown in Figure 3. The lime lump from the specimen investigated was very soft and powdery, resembling a 'pure lime lump' consistent with a lump of the original hydrated lime which was not mixed properly with the aggregate. It can therefore be concluded that the hydraulic compounds identified were due to the lime binder itself, which means that the lime binder was in fact a natural hydraulic lime.

Fig. 8 Thermogravimetric analysis.



Examination of lime plaster (Sample 18)

Results of a thermogravimetric analysis on Sample 18 are presented in Figure 8 and show a significant weight loss in the temperature range 130 to 250°C due to the dehydration of gypsum. The same procedure that was employed with Sample 14 was used to calculate the percentage of gypsum in the sample, which was 48.3%. In the manner of Sample 14, a shallow gradient was observed in the TGA curve between 200 and 600°C, which implies that some hydraulic compounds were contained within the sample. The endothermic peak at about 850°C corresponds to the decomposition of 31.5% of the total weight of calcium carbonate. The small endothermic peak at about 500°C suggests the presence of a reaction that does not affect the sample weight and, consequently, may be recognized as a phase change or the decomposition of a very small amount of organic material such as hair fibres. Dark brown hair fibres were, in fact, identified in Sample 18 during the visual analysis (Table 3).[58] In Figure 9a a low-magnification SEM image of the hair fibre is shown.

Figure 9b shows a high magnification of the lime binder that was attached to the surface of the hair, with fibular structures decorating crystals. The XRD pattern in Figure 10 shows peaks attributed to the presence of calcite at 2-theta values of 22.9, 29.1, 36.1, 39.3, 43.2, 47.3, and 48.5, suggesting that calcite crystals were present on the hair surface. Peaks at 2-theta 20.7, 31.1, and 33.3 correspond to calcium sulphate dihydrate (gypsum). The presence of gypsum in the sample is also supported by the sulphur from the EDX analysis (Figure 11). The digital images of the polished sections (Figure 12) show the presence of aggregate, which is believed to be quartz as suggested by silicon detected in the EDX spectrum.

Conclusions

The study provided useful information on the characterization of Georgian mortar and plaster, a subject that has so far received little attention. As for the limitations of the work, it is envisaged that similar research be extended to a wider number of Georgian buildings in order to have a more representative population of samples. Such work would be useful in practical terms because it could be taken as a reference when designing repair mixes.

In particular, the following conclusions can be drawn from the study.

Of the mortar samples collected and analysed a number of distinct groups were identified, containing: red brick, carbon, and white lumps; lime lumps and carbon; and carbon only. Within these, subgroups containing fibres and a finishing layer were also present.

The TGA analysis identified hydraulic compounds which can be attributed to a natural feebly hydraulic lime.

The plaster used consisted of 48.3% gypsum and 31.5% $CaCO^3$ with small quantities of C-S-H compounds present.

Analysis revealed dark brown hair in the mortars, confirming that this was used for strengthening the plaster materials.

Another important aspect that was not tackled by this research is the study of archival material as a source of recipes: their comparison to the analytical results could provide extra information on the actual mixes employed by Georgian craftsmen.

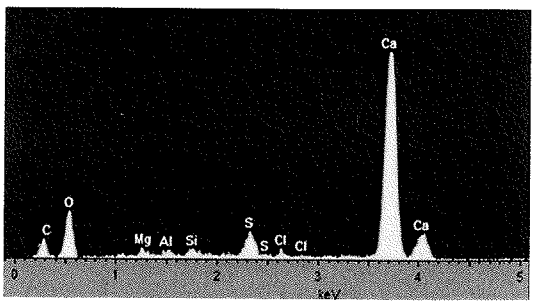


Fig. 11 EDX analysis.

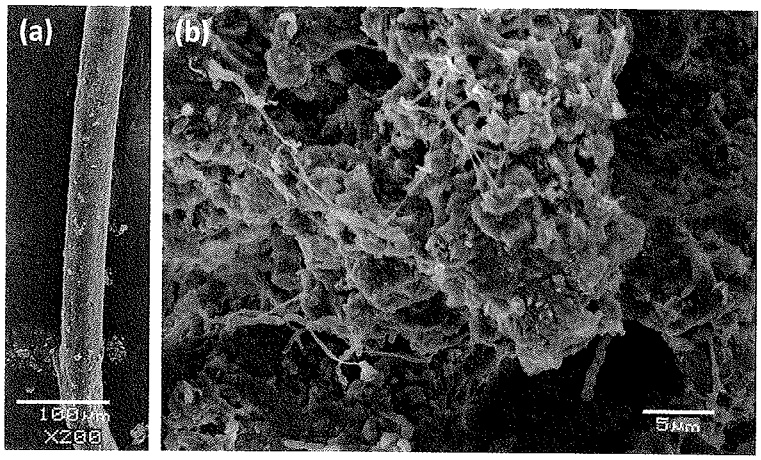


Fig. 9a Low-magnification SEM image of hair fibre.
Fig. 9b High magnification of the lime binder attached to the surface of the hair.

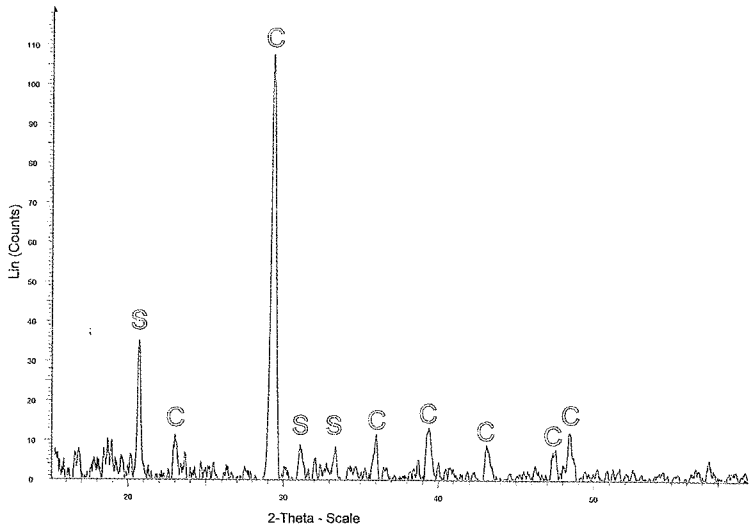


Fig. 10 XRD pattern.

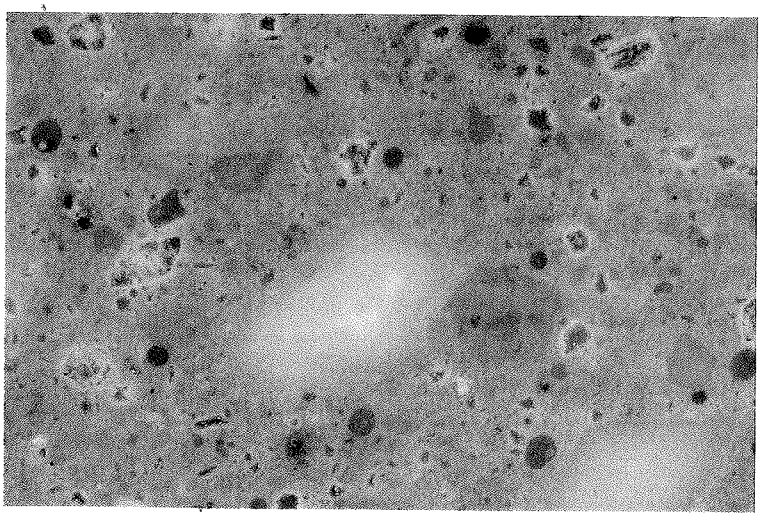


Fig. 12 Digital image of polished section showing presence of aggregate.

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